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Chapter

Perspective Chapter: Theoretical Foundations of the Extraction Process

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Abstract

The goal of the research is to create an environmentally friendly, resource-efficient technology for the production of ingredients from plant raw materials using liquefied gases. To a certain extent, this scientific research serves to fulfill the tasks specified in the decrees and decisions adopted in this direction and other normative documents: No. 63 of 27.01.2018 "Cultivation and industrialization of licorice and other medicinal plants in the Republic of Uzbekistan on the measures to further develop the processing method." Decision of the Cabinet of Ministers of the Republic of Uzbekistan, President of the Republic of Uzbekistan dated January 7, 2022 No. PF-60 "Development Strategy of New Uzbekistan for 2022-2026 and the announced Decree of the President of the Republic of Uzbekistan. "On measures to further accelerate work on systematic support of families and women" PF dated March 7, 2022 - The competition organized together with the Family and Women's Committee under the Ministry of Poverty Alleviation and Employment within the framework of priority tasks defined in Clause 5.2 of Decree No. 87. In addition, experimental studies of the process of extracting ingredients from plant raw materials using diluted carbon dioxide in the example of namatak, chamomile grapes, pumpkin and melon seeds, fruit seeds, pomegranate peel, as well as beech root (other local raw materials) will be held. Experiments were conducted to determine the influence of the main factors on the extraction of vegetable oils (extracts) based on the developed plan of experiments, mathematical models of the process were obtained as a result of mathematical-statistical processing of the experimental results, rationalization of the influencing factors parameters (pressure in the extractor, process temperature and duration) values are determined.

Keywords: extraction, biologically active substances, temperature, pressure, extraction time, ingredients from plant raw materials

1. Introduction

Solid–liquid extraction is the extraction of one or more components from a complex solid by selective solubility. Extraction – extraction from a complex solid or liquid substance of one or more of its components using a solvent with selective solubility ([1–3], pp. 520–550; [4], p. 256; [5], p. 256; [6], p. 186; [7], p. 146).

The extraction process includes the following processes: diffusion, dialysis, dissolution, desorption, osmosis, and mechanical washing.

Diffusion is the process of gradual mutual penetration of substances that border on each other.

The extraction process goes as follows:

- a. The solvent diffuses into particles of raw materials, gets to the surface of the cell through intercellular pathways, and penetrates into the cell through the cell membrane.
- b. In the interior of the cell, following desorption, biologically active substances are dissolved in a solvent.
- c. Due to unequal concentrations, dialysis occurs the transfer of ingredients from the cell through the cell wall.
- d. As a result of dialysis, a fixed diffusion part is formed on the outside of the plant material. The diffusion part is the countermeasure for extracting the ingredients, as inhibits the extraction of ingredients from the material.
- e. After passing through the diffusion part, the ingredients spread throughout the entire volume of the solvent according to the laws of free convective diffusion.

Extraction is based on experimental provided and production skills, which generalizes the concept of the influence of various factors on the extraction process ([8], pp. 11–34; [9], p. 416; [10], pp. 894–913).

The significance of extraction is explained by its ability to provide an almost exhaustive separation of dissolved components at low temperatures, which is the key to obtaining high-quality extracted substances ([3], pp. 520–550; [8], pp. 11–34). Traditional physical processing methods in oil and fat production are crushing, pressing, mixing, settling, filtering and thermal influence. Electrophysical and acoustic and other methods are considered non-traditional ([8], pp. 11–34; [11], pp. 40–42; [12], pp. 322–327).

Solvent extraction is the most widely used method in plant extraction. The principle is based on the transfer of a compound from a sample to an organic solvent ([13], pp. 169–198). These are very important operations in many industries such as chemical, biochemical, food, cosmetic, and pharmaceutical industries ([14], pp. 37–70).

Solvents used as an extractant must be selective, readily available, low cost, and harmless.

Of the common methods for extracting extracts from plant materials with solvents, the following can be cited.

Maceration describes the soaking of solid plant material in a solvent at room temperature for a specified time. Thus, the solid is simply in contact with the solvent without any movement.

Usually, the extraction time is long and the efficiency is low. This can be explained by the fact that maceration is a process in which there is an equilibrium between the concentration of the extractable component in the plant material and the extraction

solvent. This means that the extraction occurs until the equilibrium concentration of the extractable components in the solvent is reached. However, depending on the properties of the solvent, a significant amount of the desired components may remain in the plant material. Complete recovery requires repeated addition of fresh solvent, resulting in high specific solvent consumption.

If maceration is carried out at a constant elevated temperature, the method is called assimilation (**Figure 1**) ([15], pp. 310–314; [16], pp. 517–554; [17], pp. 377–387; [18], pp. 8615–8627; [19], pp. 136–137).

Disadvantages of the method: duration of the process; insufficient depletion of raw materials, low yield of biologically active substances.

A further extraction technique is infusion. Here, the solid is immersed in a heated solvent, and the process proceeds without boiling, followed by cooling of the suspension. The most striking example of an infusion is the preparation of tea. In the case of a boiling solvent, the extraction method is called decoction. However, these methods are only applicable to thermostable compounds. However, it is a very fast and sometimes unavoidable extraction method ([14], pp. 37–70).

Unlike previously described techniques, percolation is a method of exhaustive coupling by extraction from plant material. For this, solid plant material is used, and the solvent passes through the fixed layer. As a rule, this process occurs under the action of gravity from top to bottom. In addition, adequate extraction efficiency can be achieved by recycling the extraction solvent and passing it through the plant multiple times. The advantage of this method is the relatively low mechanical stress on the hard material. Moreover, additional filtration is not required as the extract contains no particulate solids. The most striking example of percolation is the preparation of coffee in a coffee machine ([14], pp. 37–70; [15], pp. 310–314). Disadvantages of the method: energy consumption when supplying the extractant to the percolator (**Figure 2**).

The percolation method includes three consecutive stages: wetting of raw materials (swelling of raw materials), infusion, and percolation itself.

Another comprehensive method for extracting compounds using a suitable solvent is the Soxhlet method.

Typically, this method is performed in a laboratory setting to determine the total amounts of compounds that can be dissolved in the solvent used. The Soxhlet installation is shown in **Figure 3**.



Figure 1.

Schematic diagram of maceration with extractant circulation. (1 - maceration tank; 2 - false perforated bottom; 3 - filter material; 4 – Pump).



Figure 2. Scheme of percolators-extractors. 1 - false bottom (perforated mesh); 2 - filtering material (burlap, linen, etc.).



Figure 3. Soxhlet laboratory setup for the extraction of plant material. 1 - Hotplate; 2 - solvent; -; 3 - Extraction thimble with sample; <math>4 - Sealing value 5 - steam riser pipe-6 - Condenser.

The previously extracted solid plant material is placed in a porous extraction thimble. It mainly consists of cellulose fibers. After that, the vessel is placed in the chamber of the Soxhlet extractor. The solvent in the lower flask is then heated to boiling. The steam vapor passes through the ascending pipe to the condenser. There, the pure solvent condenses, enters the extraction vessel, and comes into contact with the plant material. A certain amount of solvent, the chamber is emptied through a special siphon. Decomposition of components can also occur in the bottom flask, as the solvent is kept at its boiling point for several hours ([15], pp. 310–314; [16], pp. 517–554; [19], pp. 136–137; [20], pp. 169–198).

Disadvantages of the method: relative high cost of equipment, increased consumption of the extractant, high energy costs, the threat of atmospheric pollution with a volatile organic solvent.

There are various methods to speed up and improve the extraction methods described above. The use of microwaves and ultrasound as a stimulator-intensifier of the process [21].

When using ultrasound in the extraction process in the "solid-liquid" system, the main mechanism is the destruction of the surface structure of the material ([20], pp. 68–87; [22], pp. 10–23), diffusion intensification ([23], pp. 124–130), capillary sound effects, and exposure to acoustic microwaves ([24], pp. 231–255), their passage through the cells ([25, pp. 134–149) and local thermal effects ([22], pp. 10–23; [24], pp. 231–255; [26], pp. 656–659).

Disadvantages of the extraction method using ultrasound: high cost of equipment, increased energy costs, indifferent to active substances, target components.

In recent years, extraction with the help of microwaves has been intensively studied and successfully applied in the "solid-liquid" method of extraction of plant materials. Microwave radiation can be used to rapidly extract certain classes of plant compounds ([27], pp. 1–14). Microwaves directly affect the water in the plant matrix cells. These cells are destroyed by the high pressure generated by the microwaves, releasing the plant compound. The substance is dissolved in an organic solvent and then fed for processing ([14], pp. 37–70; [28], pp. 530–554; [29], pp. 708–716).

The extraction can also be carried out with a microwave-absorbing solvent. However, high heat should be avoided to prevent thermal decomposition of plant compounds ([15], pp. 310–314). Microwave extraction has a number of advantages over traditional methods. First of all, compounds can be extracted more selectively and faster. At the same time, the consumption of energy and organic solvents is reduced ([13], pp. 517–554; [14], pp. 37–70; [15], pp. 310–314; [30], pp. 105–113).

2. Features of technological parameters and factors of the extraction process

During the extraction of biologically active substances from plant materials with supercritical fluids, various individual compounds are involved, which raises the question of the effect of these or other components of the initial raw materials during the extraction process ([31], pp. 47–65; [32], pp. 101–105).

The general patterns of extraction by liquefied gases have been studied for raw materials with a variety of textures. To date, under different conditions, scientists have extracted more than a hundred different biologically active substances from plant materials, of various histological structures ([32], pp. 47–65; [33], pp. 101–105).

Factors affecting the extraction with liquefied gases are: temperature and pressure, viscosity of the solvent, grinding and moisture content of the material, infusion time, duration, and hydrodynamics of the process ([31], pp. 47–65; [33], pp. 55–62; [34], pp. 23–32). Modification of important parameters of the extraction process - provides an opportunity to obtain products of the desired composition ([35], pp. 259–262; [36], pp. 70–93; [37], pp. 50–55).

Influence of temperature and pressure. Supercritical extraction is based on diffusion processes, where the rate of the final mass transfer, in particular, is determined by the values of the diffusion coefficients at each stage, otherwise depends on their diffusion resistances. The total resistance to mass transfer ($R_{tot.}$) is the sum of internal resistance ($R_{int.}$), resistance of the diffusion layer (sub-layer) ($R_{dif. layer.}$), and convective resistance ($R_{conv.}$):

$$R_{tot.} = R_{int.} + R_{dif.layer.} + R_{conv}$$
(1)

Values $R_{dif. layer.}$ and $R_{conv.}$ (the second and third stages of extraction) are small and depend on the hydrodynamic conditions of the medium. On the efficiency of extraction at the first stage, the viscosity of the extractant, temperature, internal structure of the material, particle size of the extracted material and other factors are of decisive importance ([31], pp. 47–65).

Liquefied gases, having a low viscosity (1-2 orders of magnitude less than that of alcohol and water), excellent wetting and permeability, to a greater extent than other liquid extractants, affect the diffusion rate and solubility of the intracellular content contained in the material. This dependence is explained by the fact that with a decrease in viscosity, the diffusion coefficient (*D*) in the Einstein equation increases proportionally:

$$D = \frac{k \cdot T}{6 \cdot \pi \cdot \eta \cdot \mathbf{r}} \tag{2}$$

whereat k – Boltzmann's constant, 13805, Dj/K; T – absolute temperature, °K; π – 3,14; η – fluid viscosity, Pa·s; r – diffusing particle radius, m.

Temperature and pressure during the extraction of liquid CO_2 in saturation, determine the chemical composition and quantitative yield.

Effect of temperature: Increasing the temperature at constant pressure leads to a decrease in the density of the liquefied CO_2 , thus reducing the solubility of the solvent ([31], pp. 47–65).

With an increase in the pressure of the CO₂ extraction process, the extract yield increases.

The dependence of the increase in extraction efficiency on the increase in the temperature of the process is known, when the thermal movement of molecules is accelerated and the viscosity of the liquid components of the system decreases ([38], pp. 56–73).

Based on the position of thermodynamics that the critical point of a binary mixture is characterized by the vanishing of the first and second derivatives of the chemical potentials of the components, and from the fact that they are the driving force of isothermal molecular diffusion, the preferred development at this point is a significant slowdown in the extraction process.

Influence of the degree of grinding of raw materials. With flow extraction at a rate corresponding to the independent flow of the solvent through the plant material, the

yield of components, other things being equal, was increased by 1.5–3 times and reduced in time as a result of a decrease in the particle size of the raw material ([38], pp. 56–73; [39], pp. 80–84; [40], pp. 23–40).

The larger the contact surface of the phases, the faster the extraction. But very fine powders for extraction cannot be used:

- 1. A large amount of heavy substances, insoluble particles, and colloids pass from plant powders (destroyed cells) a cloudy liquid that is difficult to clean is obtained;
- 2. The fine powder forms a thick mass with the solvent, which resists the passage of the solvent (**Table 1**).

Influence of extraction duration. All production processes are efficient if they are short-term. The concentration of the obtained components is influenced not only by the level of crushing of the material but also by the duration of the extraction. For each type of plant material, under all other identical conditions, this value is characteristic. The study of the kinetic regularities of the extraction process with liquefied gases gives an idea of the rate of extraction of biologically active substances from plant materials.

Analyzing the dependence of the change in the content of extractable substances in the raw material on the duration of extraction, two periods of the extraction process can be distinguished. The first is the period of fast extraction, during which the process proceeds at the highest speed, and the second is the period of slow extraction, the speed of which is much lower ([40], pp. 23–40).

The time of the second extraction period is 35–40 minutes, characterized by the extraction of biologically active substances contained in microcapillaries and in the interior of intact cells. During this time, 5–10% of the components remaining in the plant material are removed. Diffusion of substances from whole cells of the material takes a long time, because of this, the duration of extraction is set by the diffusion coefficient.

Influence of persistence. The quantitative yield of the sum of extractive substances or an individual substance is influenced not only by the degree of grinding of raw materials but also by the time of the process. For each type of raw material, with all other conditions being equal, this value is individual. The low viscosity of the extractant, a large number of destroyed cells (due to the fact that in most cases the degree of grinding of raw materials is 0.25–0.50 mm) leads to the fact that with an increase in the time of infusion, not pure extractant penetrates into the cells, but a concentrated thick solution of cellular contents ([31], pp. 47–65).

Influence of humidity of extracted raw materials. The concentration of biologically active substances during extraction with liquefied gases significantly affects the moisture content of the plant material. How low the moisture content of the material, the more the concentration of the output of biologically active substances. By crushing

grass, leaves, flowers	stems, roots and bark	fruits and seeds
3–5 mm	1–3 mm	0.3–0.5 mm

Table 1.

The degree of grinding in different plant materials.

Sulfur Dioxide Chemistry and Environmental Impact

extraction time, min	The yield of e personal	xtractable substances (%) at movement speed (cm ³)
	5	10
45	3.73	3.79

Table 2.

Yield of extractable substances at different speeds.

plant material on a roller crusher with a moisture content of more than 10%, the extraction of substances from this material is difficult.

Influence of the speed of movement of the solvent on the yield of extracted substances. The significance of the coefficient of numerical mass transfer of the components is determined by the hydrodynamic conditions of the extraction process. As high as the speed of movement of the solvent, so much more concentrations of the substance pass due to convection diffusion (**Table 2**).

An analysis of the effect of the ratio of raw materials: extractant showed that the amount of fresh solvent does not have a significant effect on the residual content of extractives in the raw materials, in contrast to, for example, the degree of grinding of the raw materials.

The density of the raw material layer in the extractor is also of great importance; with an increase, the transverse unevenness of the transition of the extractant to stagnant zones increases, where the solvent either does not move at all, or moves at a low speed, which slows down the extraction in such places.

Mixing significantly reduces the thickness of the boundary layer, increases the coefficient of external diffusion, and the volumetric velocity of the liquefied gas.

The limiting factor for the widespread introduction of the extraction of plant materials with liquefied and compressed gases is the underestimation of the biological value of the products obtained and the lack of specially designed standard equipment, control systems and safety of these processes ([31], pp. 47–65).

3. Analysis of the process and design of extraction plants using CO₂

The development of methods of energy-saving technologies that allow obtaining new high-quality products in the pharmaceutical, perfumery, cosmetology and food industries is due to the acute social need for high-quality medicines and food products, as well as in environmentally friendly industries ([41], pp. 16–27).

The determining factor in the production of high-grade food products is the enrichment of their composition with the missing natural ingredients.

Analysis of scientific, technical and patent literature shows, on the one hand, the prospects of using liquefied and compressed inert gases as extractants of valuable components from vitamin-containing plant materials, on the other hand, the absence of a unified system of scientifically based use in the food industry – carbon dioxide.

In addition to technological advantages, the use of carbon dioxide as a solvent is economically beneficial, since it is a relatively cheap and readily available volatile solvent.

Despite significant advances in the field of subcritical CO_2 extraction, there is today a large gap between research and development and the speed of their implementation in industry. A number of studies have been carried out on the extraction of

biologically active components from raw materials with carbon dioxide in a pre- and supercritical state ([42], pp. 251–253; [43], pp. 26–29; [44], pp. 108–114; [45], pp. 136–148; [46], p. 324; [47], p. 207). Within the framework of the second industrialization of industry, the prospects for the extraction of target substances from raw materials with gases in various phase states are determined ([48]; pp. 368–371; [49], pp. 36–41; [50]; [51], p. 106, pp. 196–199; [52], pp. 35). The analysis of the combinatorial algorithm of the CO₂-extraction process was carried out ([53], pp. 100–102; [54], pp. 124–125; [55], pp. 217–223). Much attention is paid to the technical equipment of extraction gas-liquid enterprises ([56], pp. 1354–1365; [57], p. 445). Over the past decade, several examples of pilot large-scale projects have been reported to implement a technological and economic strategy for the production and use of CO_2 extracts ([54], pp. 124–125; [58], pp. 260–269; [59], pp. 74–77; [60], pp. 8–33; [61], p. 43).

In general, pure chemicals can be in various states of aggregation depending on pressure and temperature. On **Figure 4** shows the phase diagram of carbon dioxide (CO_2) . Using this diagram, you can understand what state a substance is in at a given pressure and temperature. In particular, there are four different regions in which the substance is in one phase: solid (t), liquid (l), gaseous (g), and supercritical (SC) ([62]; pp. 119–134).

The point at which the evaporation curve ends is called the "critical point." It is characterized by a critical temperature Tc and a critical pressure Pc ([62], p. 119–134; [63], pp. 1766–1778). The critical point of carbon dioxide is at $T_c = 31,1^{\circ}C \ \mu P_c = 7,38$ MPa ([64], [65], pp. 15–16).

For the development of extraction technology, PVT data (specific volume depending on pressure and temperature), viscosity, diffusion coefficients, solubility, density dependence on V and T are of great importance.

At 40°C, the gas density changes sharply within the limits of up to 20 MPa, it increases to 785.13 kg/m³, i.e. exceeds the density of liquid carbon dioxide (at 20°C and P = 5.9 MPa). At a pressure above 30 MPa, the gas density increases more smoothly (at 40 MPa 924.05, kg/m³, at 60 MPa 1000 kg/m³) ([66], pp. 8–12). The density of a gas depends not only on pressure but also on temperature. At a pressure of 20 MPa and a temperature of 40°C, the gas density is ρ = 785.13 kg/m³, at 100°C - ρ = 481.3 kg/m³, at 200°C - ρ = 258.15 kg/m³ (**Figure 4**).



Figure 4. *Phase diagram of carbon dioxide.*

Supercritical extraction technology finds its application in food, cosmetics and pharmaceutical industries ([64], [67], pp. 977–982; [68, 69], pp. 1–106.).

Extraction with supercritical carbon dioxide is used to extract volatile (e.g. essential oil) and/or non-polar (e.g. fats, waxes) compounds from plant materials. There are many variables that must be considered in order to optimize the extraction yield and selectivity of supercritical extraction ([29], pp. 708–716; [70], pp. 771–782; [71], pp. 2–24; [72], pp. 1–37). However, in most cases of extraction from plant materials, the diffusion of the target compound from the matrix is usually the rate-limiting step ([73], pp. 320–325). This can be avoided either by increasing the temperature and hence the volatility of the compound, or by using high solvent densities. To achieve good selectivity of SC extraction, the density of the solvent must be carefully controlled ([16], pp. 517–554; [70], pp. 771–782; [74], [75], pp. 1–15, [76], pp. 143–149, [77], pp. 1–5; [78], pp. 58–69; [79], pp. 27–51).

Modifiers are used to improve the extraction ability of supercritical – CO₂, among which methanol is the most commonly used co-solvent for supercritical extraction due to its polar properties and good miscibility with CO₂ ([70], pp. 771–782). Longer extraction times increase the extraction yield of most compounds. A distinction should be made between static and dynamic extraction. It has been shown that a preliminary static extraction step can increase the yield as the contact of the sample with the solvent is improved ([71], pp. 2–24; [76], pp. 143–149). The flow rate of supercritical carbon dioxide through the extraction vessel also has a strong influence on the extraction efficiency ([71], pp. 2–24; [72], p. 1–37). Reducing the particle size of the plant material leads to an increase in surface area and facilitates the process. However, excessive grinding of the solid can make extraction difficult due to readsorption of the target compounds on the surface of the matrix. ([71], pp. 2–24; [76], pp. 143–149; [80], pp. 22–26).

Our country is very rich in plant raw materials for the production of food biologically active substances, pharmaceutical substrates, as well as perfumery raw materials. But so far no thorough research has been conducted on this topic in Uzbekistan.

Improving the technique and technology of extraction of plant raw materials with carbon dioxide is possible on the basis of research on both the extraction process itself, focused on local raw materials, and the operation of the entire complex of apparatuses of the extraction plant is very important in terms of obtaining high-quality products at a relatively low cost ([81], pp. 87–95).

The criteria for choosing a new extraction technology with liquefied CO_2 are: separation by traditional methods is impossible or expensive or unsatisfactory; – at least one of the properties of liquefied and compressed gases solves the problem, the value of the resulting target product justifies the economic costs.

Liquid CO_2 does not support the vital activity of microorganisms and molds, which makes it possible to obtain sterile products even when using raw materials contaminated with microorganisms, liquid CO_2 is thermally stable at normal temperatures, and is chemically inert. The separation of the solvent from the extract is possible either by lowering the pressure or by heating, which converts liquid CO_2 into a gaseous state, while CO_2 is released - extracts ([80], pp. 22–26).

The use of liquefied carbon dioxide as an environmentally friendly solvent is one of the rapidly developing areas in the development of the latest state-of-the-art gasliquid technologies.

In the supercritical state, gases are substances used as solvents at temperatures and pressures exceeding the critical value. Many gases have a critical temperature close to

room temperature and a critical pressure in the range of 5.0–8.0 MPa, which makes them very convenient and inexpensive to use in industry.

The extensive formation of technologies using supercritical gases is associated with the use of their unique properties. This is a combination of the properties of gases at high pressures (low viscosity, high diffusion coefficient) and liquids (high dissolving power). With this, the dissolving power of supercritical gases is very sensitive to changes in pressure or temperature ([82], pp. 47–49).

Very prominent is the possibility of carrying out a rapid mass transfer, carried out due to the low viscosity and high diffusion coefficient; the combination of low interfacial intergrowth with low viscosity and high diffusion coefficient, which makes it easier for supercritical gases to penetrate porous media compared to liquid solvents. The main condition is the easy decomposition of liquefied gases and substances extracted in them by pressure reduction.

Depending on the method of phase contact, extractors can be divided into three groups: stepped or sectional, differential-contact, and mixing-settlement ([5], p. 256; [83], p. 388).

- In international technological practice, installations for carrying out CO₂ extraction are known, which can be cited as an example:
- for extraction with liquefied gases at doktric parameters of pressure and temperature;
- for extraction with liquid gases at a pressure above and a temperature below the critical one;
- for extraction with compressed supercritical gases;
- with mixed processes multi-stage ([60], pp. 8–33; [84], pp. 21–26; [85], pp. 394–399; [86], pp. 166; [87], pp. 388–389).

At present, CO₂ extraction plants have a number of disadvantages. For example, in the extraction shop of the Yuventa company (Krasnodar), extraction modules manufactured in the last century were installed. Part of the equipment of the modules is not made of stainless steel but of ferrous metal. There is no installation for mixing raw materials with solvents ([46], p. 324; [88], pp. 43–46).

The most advanced equipment is installed in the extraction shop of Caravan LLC (Krasnodar). It provides for the delivery of liquid carbon dioxide in tank trucks with a capacity of up to 8 tons, high-pressure extractors with a capacity of up to 150 liters, and the possibility of quickly removing CO₂ vapor from the evaporator to the condenser. The disadvantage of the workshop equipment is an irrational system for loading and unloading raw materials into extractors ([89], p. 152; [90], pp. 94–97).

The universal extraction module of Caravan Company LLC [91] is designed for the extraction of biologically active substances from plant raw materials with liquid CO₂.

In the experimental plant of the Research Institute "Mir-Prod-Mash" (Moscow), extracting modules were produced, which include 2 extractors of 10 liters each, an evaporator and a condenser. However, the design of the module does not provide for intensive mass transfer in the "solid-liquid" system; as a result, the extraction of components from the raw material lasts 8 hours.

The formation of effective equipment samples is associated with the primary development of extraction modes and designs of individual units at laboratory facilities.

A schematic diagram of the installation for the extraction of ingredients from tea and medicinal raw materials with liquid CO_2 is shown in **Figure 5**.

A feature of the improved laboratory setup is the inclusion of a vacuum pump 13 in the circuit, which allows air to be evacuated from the extractor and plant material weighed (**Figure 6**). Placement of miscello-receivers outside the body of the sealed apparatus makes it possible to select fractions of extractive substances depending on the duration of the process of extracting plant materials.

Distinctive features include lining the internal surfaces of equipment with ceramic, metal-ceramic and carbon-plastic coatings, improving the drainage system of extractors due to drainage ceramic tubes, increasing the heat exchange surface of the evaporator and condenser ([89], p. 152; [90], pp. 94–97).

Figure 6 shows the hardware-technological scheme for the production of CO_2 extracts, created in the conditions of the extraction shop of Caravan Company LLC (**Figure 7**).

The developed devices ensure the achievement of results in the field of CO₂ - the extraction of significant ingredients from plant material.

The patterns of interaction, functioning and development of technological processes based on the interaction of food products with CO_2 in stable or changing phase states have been established, which made it possible to develop the scientific foundations of engineering solutions in the field of engineering and technology of CO_2 - processing of plant material [92].



Figure 5. *Schematic diagram of the installation for extraction with liquid carbon dioxide.*



Figure 6. *Improved batch extraction plant.*



Figure 7. *Hardware-technological scheme for the production of* CO_2 *extracts.*

Apparatus and equipment for carbon dioxide extraction are classified in the following works ([79], pp. 27–51; [92], [93], p. 495; [94], [95], p. 390).

The following scientific study describes a multi-stage (subcritical / supercritical) pilot plant (**Figure 8**) from Separeco S.r.l. (Italy) ([96], pp. 358–363).



Figure 8.

Combined scheme of sub- and supercritical extraction of valuable components from plant materials.



Figure 9. Scheme of the modernized installation for studying the process of CO_2 extraction in the pre- and supercritical state of carbon dioxide.

According to the principle of operation, the devices can be divided into laboratory, experimental (desk or pilot) and industrial ([97], pp. 6–42; [98], p. 240).

The scientific work [98] describes in detail the process of assembling an apparatus for supercritical fluid extraction, created and manufactured at the Technical University Hamburg Harburg (TUHH) ([99], pp. 210–247).

In the construction of the following work, a greater porosity and porosity of plant raw materials is achieved, which contributes to a higher product yield and intensifies the process, reducing the extraction time [90, 91].

And the following study shows the installation (**Figure 9**) equipped with a device for superheating the solvent and a high-pressure pump, which makes it possible to extract the ingredients sequentially in the sub- and supercritical states of the solvent ([100], pp. 13–21).

Extracts obtained from plant materials using carbon dioxide are used in the food, perfume and pharmaceutical industries, as they are more environmentally friendly than extracts extracted by other traditional methods.

4. Licorice root and its chemical composition

Currently, reforms are being carried out in Uzbekistan in the field of protection of medicinal plants, rational use of natural resources, creation of plantations for growing medicinal plants and their processing [101].

In Uzbekistan, 4.3 thousand plants, of which 750 varieties are medicinal, 112 varieties are registered for use in scientific medicine, 70 species are used in the pharmaceutical industry.

By presidential decree No. 4670, reproductive organs, that is, seedlings, typical gray soils suitable for flat lands, moisture-loving, saline (cryptohalophytic) plant - licorice or licorice root (Glycyrrhiza glabra L.) are recommended for planting in all regions of the Republic of Karakalpakstan, Syrdarya, Khorezm areas [101].

There are about 13 types of licorice root (licorice) in the world. The most common species are: licorice naked (Glycyrrhiza glabra), Ural licorice (Glycyrrhizauralensis Fisch) and Korzhinsky licorice (Glycyrrhizae KorshinskyiGrig). Licorice is the most popular among them, its roots contain the largest amount of biologically active substances (BAS) ([102], pp. 11–13; [103], p. 314).

Licorice root is a rather high-calorie product, 100 g contains 375 kcal. It contains no proteins, almost no fat (0.05 g), a small amount of fiber (0.2 g) and a lot of carbohydrates (94 g). Dried licorice roots have been widely used since ancient times as a food flavoring agent, sweetener, and for various medicinal purposes [104, 105].

The art of healing with the help of licorice root extracts, developed by Eastern medieval medicine, is summarized by Avicenna. Avicenna considered the nature of the licorice root to be balanced ([106], pp. 150–155).

Licorice root, as well as biologically active substances isolated from them, has a wide range of pharmacological properties ([107], pp. 13–26). To date, the results of more than 2 thousand scientific studies on the licorice root and its biologically active substances have been published, which confirms the great interest of pharmacologists in plants of this genus in many countries of the world. To date, licorice root in all countries is widely used in the food, pharmacological, cosmetology industries ([108], p. 210; [109], pp. 189–191; [110], pp. 108–114; [111], pp. 21–27).

Not to mention the fact that interest in licorice root has grown incredibly sharply over the past 20 years and it is in first place among herbal preparations ([112], p. 378).

Pharmacological and chemical properties of licorice root have been comprehensively studied by I. A. Muravyov, K. Z. Zakirov, V. I. Litvinenko ([113], p. 191).

In ([102]; With. 11–13) for the first time studied the chemical composition of the roots of licorice growing in the Samara region. Glycyrrhizic acid (triterpene saponin) and flavonoids - licurazid, liquiritin, liquiritigenin (flavonones) were isolated and identified from the roots of the licorice root using UV, 1H-NMR spectroscopy, and mass spectrometry, the results of chemical transformations; isoliquiritin, isoliquiritigenin (chalcones); ononin, formononetin (isoflavones).

A processed product, that is, licorice root extract, is very popular. Experts are unanimous that the global demand for finished products made from licorice root will constantly increase ([114], p. 466).

The scientific literature describes about 80 triterpenoids and over 300 individual phenolic compounds, several dozen polysaccharides, amino acids and many other substances that have a variety of pharmacotherapeutic properties found in the licorice root ([107], pp. 13–26; [115], pp. 16–19; [116], p. 83; [117], pp. 152–158; [118], pp. 1954–1969).

The main active ingredients and biologically active components of licorice root are triterpene glycosides (the most important of them is glycyrrhizic acid), the content of which can reach 25% by weight of dry material, various phenolic compounds, which account for 3–5%, glabridin and carbohydrates. The total content of extractive substances can reach 40% ([119], pp. 55–59).

The quality of the licorice root is standardized by the State Pharmacopeia (GF): the content of extractable components extracted by 0.25% ammonium hydroxide solution must be at least 25%, moisture not more than 14%, ash not more than 8%, glycyrrhizic acid – not less than 6% ([107], pp. 13–26; [120], pp. 11–13).

The licorice root contains biologically active substances such as: flavonoids up to 5.0%, carbohydrates up to 34.0%, proteins - up to 10.1%, amino acids up to 12.71%, including asparagine up to 4.0%, lipids up to 4.7%, vitamin C up to 3.1% ([107], pp. 13–26; [121], p. 175).

Glycyrrhizic acid with high-intensity sweetness (about 50 times more than sucrose), widely used in all countries as a biological additive in the food, beverage, cosmetic, pharmaceutical and tobacco industries ([122], p. 271; [123], pp. 55–72). The content of glycyrrhizic acid in licorice roots varies in the range of 2–24% ([124], pp. 166–168). Glycyrrhizic acid is highly soluble in ethanol and hot water, insoluble in cold ([120], pp. 145–158; [125], pp. 87–94; [126], pp. 32–34). Melting point (Tm) of glycyrrhizic acid is 220° C). The UV spectrum of glycyrrhizic acid shows that its maximum absorption peak is in the region of 254 nm ([127], p. 1331; [128], pp. 100–104; [129], pp. 87–91).

Glycyrrhizic acid was first extracted from the licorice root by the French scientist Pierre Jean Robiquet in 1809 and gave the name glycyrrhizin. After Z. Russin named glycyrrhizic acid in 1876 (**Figure 10**) ([107], pp. 13–26).

Glycyrrhizic acid is active against a wide range of viruses, including herpes, corona, alpha, and flaviviruses, human immunodeficiency virus, type I poliovirus, vesicular stomatitis virus, and influenza A virus ([128], pp. 100–104; [130], pp. 1256–1259; [131], p. 333; [132], pp. 199–206).

Nowadays, the following phenolic compounds have been isolated and established from the licorice root: simple phenols (phenol, resorcinol, pyrogallol), phenolcarboxylic acids, hydroxycinnamic acids (ferulic acid, cinnamic acid, synapic acid).

To date, over 50 different flavonoids have been extracted and identified from licorice root. ([131], p. 333; [133], pp. 6–8; [134], pp. 34–80; [135], pp. 1027–1030).



Structural formula of glycyrrhizic acid.

The content of flavonoids in licorice roots is 3–6%. The work 136 presents the main flavonoids of liquorice root and their characteristics ([134], pp. 34–80).

Carbohydrates extracted from licorice root: established ([124], pp. 166–171; [135], pp. 1027–1030) that the carbohydrate content in the licorice root reaches up to 40%.

In ([136], pp. 10–24), the author studied the properties and chemical composition of various morphological organs of liquorice collected from the northern regions of the Republic of Uzbekistan. The chemical composition of licorice root is presented in **Table 3**.

Traditionally, licorice root is most actively (due to climatic features) grown in the north-west of Uzbekistan - in Karakalpakstan and the Khorezm region [137].

The president of the Japanese company, Naomasa Yoshida, emphasizes that the laboratories of Cokey Systems Co. Ltd. regularly test samples of licorice root from many countries, while licorice root from Uzbekistan is recognized as the best in the world in terms of its composition, thickness, smell and color and other quality attributes [138].

In Ref. ([139], pp. 1338–1340), the chemical characteristics of licorice root collected from Uzbekistan are given. A study of Glycyrrhiza glabra L. was carried out in Uzbekistan in August 2001 and above and below ground parts of the plant were collected from a site near Yangiyer, 100 km south of Tashkent. Licorice, which grows in Uzbekistan, contains glycyrrhizin up to 6.1% of dry weight.

In the study ([140], pp. 677–680), the author also identified flavonoids glycionide A and B - glucose (up to 15.2%), sucrose (up to 11%), starch, resinous substances,

Name of substances	Content, %
Substances extractable with a mixture of alcohol / benzene	13.45
Easily hydrolysable substances	11.20
Substances that are difficult to hydrolyze	57.60
Reducing sugars	15.50
Cold water extractable	44.90
hot water extractable	49.40
Cellulose according to Kirchner	29.60
Nitrogen	0.97

Table 3.Chemical composition of licorice root.

Name of substances	Content, %
Extractives	22.8-44.1
Triterpenoids	7.3–23.6
Carbohydrates (glucose, sucrose, starch)	18.2–34.0
Flavonoids	3.0-4.0
Steroids	1.5–2.0
Ascorbic acid	1.1–3.1
Essential oils	1.5–2.0
Asparagine	1.0-4.0
Resinous substances	1.7–4.1
Fats and fat-like substances	0.2–4.7
Protein	6.2–10.1
Comedy	1.5–6.5
Bitterness insoluble (in water)	1.8–4.0
Ash (total)	4.9–9.7

Table 4.

The main biologically active substances of licorice root.

gums. There are many organic acids in the licorice root – salicylic, synapic, ferulic, caffeic and others. Coumarins, alkaloids, tannins, steroids, estradiol, vitamins C, B were also determined. K, Ca, Fe, Si, Sn salts are concentrated in the licorice root.

According to the literature, licorice root contains a number of biologically active substances, such as triterpene saponins, flavonoids, coumarins and other phenols, in accordance with **Table 4** ([141], pp. 30–51; [142], pp. 1868–1886). The total volume of extractive biologically active substances isolated from licorice roots reaches 40% of the mass of the feedstock ([143], pp. 55–59).

Most medicinal products based on licorice root require the introduction of modern methods of quality control into the regulatory documentation for the standardization of pharmacological substances of plant materials "Licorice Roots" ([107], pp. 23–31).

Development phase	Biologically active substances								
	glycyrrhizin acid	sugar	flavonoids						
regrowth	$\textbf{7.67} \pm \textbf{1.30}$	$\textbf{7.80} \pm \textbf{2.12}$	$\textbf{1.15}\pm\textbf{0.50}$						
	8.49 ± 1.35	$\textbf{4.92} \pm \textbf{1.50}$	3.60 ± 0.22						
bloom	10.82 ± 1.81	9.19 ± 1.05	$\textbf{3.41}\pm\textbf{0.85}$						
	6.85 ± 1.60	5.64 ± 1.00	0.92 ± 1.00						
fruiting	14.76 ± 0.50	$\textbf{6.65} \pm \textbf{1.40}$	2.62 ± 0.46						
	$\textbf{10.96} \pm \textbf{2.00}$	8.59 ± 0.35	1.05 ± 0.35						

Table 5.

The content of glycyrrhizic acid, sugars, flavonoids in licorice roots depending on the vegetation phase, % of absolutely dry weight.

regrowth	Ν	Р	К	Ca	Mg	Cu	Mn	Zn
		%		g/l	ĸg		mg/kg	
bloom	3.38	0.23	0.32	12.37	5.22	9.63	23.50	20.63
	2.67	0.23	0.39	12.64	4.54	6.13	11.50	14.50
fruiting	3.08	0.32	0.35	13.62	1.91	5.56	14.75	20.50
	2.88	0.38	0.39	14.02	0.84	4.69	9.63	11.25
Table 6.	1 [[\bigcap	$\left[\right]$				

The content of mineral elements in licorice root, in terms of absolute dry weight (averaged data).

A solid extract is obtained by further evaporation of the thick extract to a solid state ([102], pp. 11–13; [144], [145], pp. 35–41). **Tables 5** and **6** show the characteristics of licorice root.

In the licorice root, an important structure is glycyrrhetinic acid, which is found in a natural compound – glycyrrhizin.

The main pentacyclic triterpenoid isolated from licorice root extracts, glycyrrhetic (glycyrrhetic) acid, is active against test microbes of staphylococcal, intestinal, and spore-forming groups ([146], p. 311). Glycyrrhetic acid has shown a protective effect against *C. albicans* fungal infection in mouse models ([147], pp. 310–315), as well as staphylococcal pneumonia caused by bacteria *Staphylococcus aureus* (**Figure 11**) ([148]; pp. 201–206; [149], pp. 241–248).

The antibacterial activity of sulfonamide GLA derivatives against gram-positive (*S. aureus*, Bacillus anthracis, Corynebacterium bovis) and gram-negative (*Klebsiella pheumonie*, *Proteus vulgaris*, *Escherichia coli*) bacteria has been established ([146], p. 311). However, the use of glycyrrhetic acid as a basic structure (scaffold) for the synthesis of its biologically active derivatives, as well as the study of the structure–activity dependence, is still a poorly studied direction ([148], pp. 201–206).

Phenolic compounds of licorice root have been sufficiently studied and widely presented in domestic and foreign publications ([147], pp. 1–158; [150], pp. 705–709; [151], pp. 4267–4272). Interest in licorice flavonoids is associated with their biological properties used in the production of medicines, food, technical and other products [152]. Interest in licorice flavonoids has especially increased over the past 20 years ([107], pp. 13–26; [116], p. 83; [153], pp. 7408–7414).

Essential oil (0,03%) ([154], pp. 5–14), it contains aldehydes, ketones, alcohols and their derivatives: ([155], pp. 1238–1241; [156], pp. 1179–1182); organic acids and



Figure 11. Structural formula of glycyrrhetic acid.

macro elements, mkg/g micro elements, mkg/g																
К	Ca	u Mn	Fe	Mg	Cu	Zn	Cr	Al	Ba	V	Se	Ni	Sr	Pb	В	
14.	5 11.	5 2.4	0.7	0.15	0.3	0.33	0.07	0.5	0.4	0.3	12.1	0.6	1.01	0.03	54.8	

Table 7.

Macro- and microelements of licorice root.

their derivatives: propionic, phenylpropionic, caproic, caprylic, pelargonic acids and other compounds ([157], pp. 1179–1182); aromatic compounds ([154], pp. 5–14), tetramethylpyrazine ([156], pp. 1179–1182); higher aliphatic hydrocarbons: tetradecane; esters of higher fatty acids: ethyl talmitate, ethyl linoleate, ethyl linolenoate ([155], pp. 1238–1241); phenolcarboxylic acids and their derivatives: ferulic, synapic ([153]; p. 193), salicylic, salicylic acid acetate ([158], pp. 259–269); higher aliphatic hydrocarbons and alcohols (in the hydrolyzate): nonacosan, tetracosanol, octacosanol ([159], pp. 620–621); higher fatty acids (in the hydrolyzate): palmitic, oleic, etc. ([107], pp. 13–26; [160], pp. 159–162).

Licorice roots contain ash – 7.88%. **Table 7** shows the macro and micro elements of the root.

Licorice root is widely used in the food industry - extracts, syrups, as a sugar substitute and foam-forming drug in soft drinks (licorice extract is one of the main parts of Coca-Cola and Pepsi-Cola), beer, kvass, many drinks, are used in food preparation such as: coffee, cocoa, marinades, compotes, kissels, flour and whipped products, sweets, halvahs. They are added as a flavor additive - in the production of fish and as a bioadditive to long leaf and green tea. In Kyrgyzstan, they replace tea. In Japan – as a dietary antioxidant supplement; in Japan and Egypt – among component additives with bactericidal and fungicidal properties for food and drinks [157].

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